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**Soil quality — Determination of  
particle size distribution in mineral  
soil material — Method by sieving and  
sedimentation**

*Qualité du sol — Détermination de la répartition granulométrique  
de la matière minérale des sols — Méthode par tamisage et  
sédimentation*

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

This document was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical and physical characterization*.

This third edition cancels and replaces the second edition (ISO 11277:2009), which has been technically revised. The main changes compared to the previous edition are as follows:

- Alternative digestion methods were added;
- A practical order of preparation steps was added;
- References were updated;
- Document has been editorially revised.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

## Introduction

The physical and chemical behaviour of soils is controlled in part by the amounts of mineral particles of different sizes in the soil. The subject of this document is the quantitative measurement of such amounts (expressed as a proportion or percentage of the total mass of the mineral soil), within stated size classes.

The determination of particle size distribution is affected by organic matter, soluble salts, cementing agents (like iron compounds), relatively insoluble substances such as carbonates and sulfates, or combinations of these. Some soils change their behaviour to such a degree, upon drying, that the particle size distribution of the dried material bears little or no relation to that of the undried material encountered under natural conditions. This is particularly true of soils rich in organic matter, those developed from recent volcanic deposits, some highly weathered tropical soils, and soils often described as “cohesive” (see Reference [4]). Other soils, such as the so-called “sub-plastic” soils of Australia, show little or no tendency to disperse under normal laboratory treatments, despite field evidence of large clay content.

The procedures given in this document recognize these kinds of differences between soils from different environments, and the methodology presented is designed to deal with them in a structured manner. Such differences in soil behaviour can be very important, but awareness of them depends usually on local knowledge. Given that the laboratory is commonly distant from the site of the field operation, the information supplied by field teams becomes crucial to the choice of an appropriate laboratory procedure. This choice can be made only if the laboratory is made fully aware of this background information.

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# Soil quality — Determination of particle size distribution in mineral soil material — Method by sieving and sedimentation

**WARNING** — Persons using this document should be familiar with usual laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

**IMPORTANT** — It is absolutely essential that tests, conducted in accordance with this document, be carried out by suitably qualified staff.

## 1 Scope

This document specifies a basic method of determining the particle size distribution applicable to a wide range of mineral soil materials, including the mineral fraction of organic soils. It also offers procedures to deal with the less common soils mentioned in the introduction. This document has been developed largely for use in the field of environmental science, and its use in geotechnical investigations is something for which professional advice might be required.

A major objective of this document is the determination of enough size fractions to enable the construction of a reliable particle-size-distribution curve.

This document does not apply to the determination of the particle size distribution of the organic components of soil, i.e. the more or less fragile, partially decomposed, remains of plants and animals. It is also realized that the chemical pre-treatments and mechanical handling stages in this document could cause disintegration of weakly cohesive particles that, from field inspection, might be regarded as primary particles, even though such primary particles could be better described as aggregates. If such disintegration is undesirable, then this document is not used for the determination of the particle size distribution of such weakly cohesive materials.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

ISO 11265, *Soil quality — Determination of the specific electrical conductivity*

ISO 11464, *Soil quality — Pretreatment of samples for physico-chemical analysis*

## 3 Terms and definitions

There are no normative references in this document.

## 4 Symbols

The following symbols are found throughout the text and, where appropriate, units and quantities are as given below (the SI convention is followed for common units, e.g. g = gram; m = metre; mm = millimetre; s = second, etc.).

Mg megagram ( $10^6$  g)

## 5 Principle

The particle size distribution is determined by a combination of sieving and sedimentation, starting from air-dried soil (see Reference [4]). A method for undried soil is given in Annex A. Particles not passing a 2 mm aperture sieve are determined by dry sieving. Particles passing such a sieve, but retained on a 0,063 mm aperture sieve, are determined by a combination of wet and dry sieving, whilst particles passing the latter sieve are determined by sedimentation.

The pipette method is preferred. As an alternative, the hydrometer method is given in Annex B. A combination of sieving and sedimentation enables the construction of a continuous particle-size-distribution curve.

The key points in this procedure are summarized as a flow chart in Figure 2. This document requires that the proportions of fractions separated by sedimentation and sieving be determined from the masses of such fractions obtained by weighing. Other methods of determining the mass of such fractions rely on such things as the interaction of particles with electromagnetic radiation or electrical fields (see Reference [2]). There are often considerable difficulties in relating the values obtained by these different methods for the same sample. It is one of the intentions of this document that close adherence to its details should help minimize interlaboratory variation in the determination of the particle size distribution of mineral soils. Therefore, the proportions of fractions shall be determined only by weighing. If this is not the method used, then conformance with this document cannot be claimed in the test report (see Clause 10).

Both the pipette and hydrometer methods assume that the settling of particles in the sedimentation cylinder is in accordance with Stokes's Law (see References [2],[4], and [7]), and the constraints that this implies, namely:

- a) the particles are rigid, smooth spheres;
- b) the particles settle in laminar flow, i.e. the Reynolds Number is less than about 0,2; this constraint sets an upper equivalent spherical particle diameter (see below) slightly greater than 0,06 mm for Stokesian settling under gravity (Reference [2]);
- c) the suspension of particles is sufficiently dilute to ensure that no particle interferes with the settling of any other particle;
- d) there is no interaction between the particle and fluid;
- e) the diameter of the suspension column is large compared to the diameter of the particle, i.e. the fluid is of "infinite extent";
- f) the particle has reached its terminal velocity;
- g) the particles are of the same relative density.

Thus, the diameter of a particle is defined in terms of the diameter of a sphere whose behaviour in suspension matches that of the particle. This is the concept of *equivalent spherical diameter*. It is the principle upon which the expression of the diameter of particles, as derived from sedimentation, is based in this document.

Stokes's Law can be written, for the purposes of this document, as in [Formula \(1\)](#):

$$t = 18\eta h / [(\rho_s - \rho_w) g d_p^2] \quad (1)$$

where

- $t$  is the settling time, in seconds, of a particle of diameter  $d_p$  (see below);
- $\eta$  is the dynamic viscosity of water at the test temperature (see [Table B.2](#)), in millipascals per second;
- $h$  is the sampling depth, in centimetres;
- $\rho_s$  is the mean particle density, in megagrams per cubic metre (taken as 2,65 Mg/m<sup>3</sup>; see note);
- $\rho_w$  is the density of the liquid containing the soil suspension, in megagrams per cubic metre (taken as 1,00 Mg/m<sup>3</sup>; see note);
- $g$  is the acceleration due to gravity, in centimetres per second squared (taken as 981 cm/s<sup>2</sup>);
- $d_p$  is the equivalent spherical diameter of the particle of interest, in millimetres.

NOTE 1 It is realized that there are considerable differences between the densities of soil particles, but for the purposes of this document it is assumed that the mean particle density is that of quartz, i.e. 2,65 Mg/m<sup>3</sup> (Reference [8]), as this is the commonest mineral in a very wide range of soils. The density of water is 0,998 2 Mg/m<sup>3</sup> and 0,995 6 Mg/m<sup>3</sup> at 20 °C and 30 °C, respectively (see Reference [6]). Given the effect of the addition of a small amount of dispersant (see [9.3.2](#)), the density of water is taken as 1,000 0 Mg/m<sup>3</sup> over the permitted temperature range of this document (see [9.2.2](#)).

Furthermore, for routine use, it is recommended that the sampling times be converted to minutes and/or hours, as appropriate, to lessen the risk of error (see [Table 3](#)).

Particles within particular size ranges or classes are commonly described as cobbles, gravel, coarse sand, silt, etc. The meaning of such trivial names differs between countries, and in some cases there are no exact translations of such words from one language to another; for example, the Dutch word “zavel” has no equivalent in English. The only fraction for which there appears to be common agreement is clay, which is defined as material of less than 0,002 mm equivalent spherical diameter (References [4]). Such trivial names shall not be used in describing the results of particle size determination according to this document. Phrases such as “... passing a 20 mm aperture sieve ...” or “... less than 0,063 mm equivalent spherical diameter ...” shall be used instead. If trivial names shall be used, for example, to cross-reference to another International or National Standard, then the trivial name should be defined explicitly, so as to remove any doubt as to the meaning intended, e.g. silt (0,063 mm to 0,002 mm equivalent spherical diameter) (see Clause 4). Furthermore, it is common to use the word “texture” to describe the results of particle-size-distribution measurements, e.g. “the particle size of this soil is of clay texture”. This is incorrect as the two concepts are different, and the word “texture” shall not be used in the test report (see Clause 10) to describe the results obtained by the use of this document.

## 6 Field sampling

The mass of sample taken in the field shall be representative of the particle size distribution, especially if the amount of the larger particles is to be determined reliably. [Table 1](#) gives recommended minimum masses.

**Table 1 — Mass of soil sample to be taken for sieving**

Maximum size of material forming > 10 % of the soil (given as test sieve aperture, in mm)	Minimum mass of sample to be taken for sieving kg
63	50
50	35
37,5	15
28	6
20	2
14	1
10	0,5
6,3	0,5
5	0,2
2 or smaller	0,1

## 7 Sample preparation

Samples shall be prepared in accordance with the methods given in ISO 11464.

NOTE For many purposes, particle size distribution is determined only for the fraction of the soil passing a 2 mm aperture sieve. In this case, the test sample (9.5) can be taken either according to the procedures in ISO 11464 or from the material passing a 2 mm aperture sieve according to 8.2.

## 8 Dry sieving (material >2 mm)

### 8.1 General

The procedure specified in this clause applies to material retained on a 2 mm aperture sieve. Table 2 gives the maximum mass which shall be retained on sieves of different diameters and apertures. If more than this amount of material is retained, then it shall be subdivided appropriately and sieved again.

It is common to refer to sieves as having a particular mesh-size or mesh number. These are not the same as the sieve aperture, and the relationship between the various numbers is not immediately obvious. The use of mesh numbers as a measurement of particle size is difficult to justify, and shall not be used in reporting the results of this document.

### 8.2 Apparatus

**8.2.1 Test sieves**, with apertures according to ISO 565, and with well-fitting covers and receivers.

The full range of sieves appropriate to the largest particle(s) present should be used (see Table 1 and 9.2.3). The apertures chosen shall be stated in the test report (Clause 10). The accuracy of the sieves shall be verified monthly against a set of master sieves kept for this purpose, using an accepted method such as particle reference materials, microscopy, etc. (see Reference [2] depending on the sieve aperture. Tolerances shall meet the requirements of ISO 3310-1 and ISO 3310-2. Sieves that do not meet these specifications shall be discarded. A record shall be kept of such testing.

Brass sieves are particularly liable to splitting and distortion, and steel sieves are strongly recommended for the larger apertures.

Special care shall be taken to ensure that covers and receivers do not leak. Sieves shall be inspected weekly when in regular use, and on every occasion if used less often. A record shall be kept of such inspections. Round-hole sieves shall not be used.

**8.2.2 Balance**, capable of weighing to an accuracy of within  $\pm 0,5$  g.

**8.2.3 Mechanical sieve shaker.**

It is usually impracticable to sieve mechanically at sieve apertures much greater than 20 mm, unless very heavy-duty equipment is available. Mechanical sieve shaking is essential to sieve efficiency at smaller apertures.

**8.2.4 A sieve brush and a stiff brush.**

**8.2.5 Ultrasonic bath.**

### 8.3 Procedure

Weigh the dry test sample, prepared in accordance with ISO 11464, to the nearest 0,5 g ( $m_1$ ). Place the weighed material on the 20 mm sieve, and by brushing the material gently over the sieve apertures with the stiff brush (to remove any adhering soil), sieve the material. Take care not to detach any fragments from the primary particles. Sieve the retained material on the nest of sieves of selected apertures (8.2.1) and record the amount retained on each sieve to the nearest 0,5 g. Do not overload the sieves (see Table 1) but sieve the material in portions if necessary.

Weigh the material passing the 20 mm aperture sieve ( $m_2$ ), or a suitable portion of it ( $m_3$ ) (see Table 2) obtained by an appropriate subsampling method (see Clause 6), and place this on a nest of sieves, the lowermost having an aperture of 2 mm. Shake the sieves mechanically until no further material passes any of the sieves (see Note). Record the mass of material retained on each sieve and the mass passing the 2 mm aperture sieve.

The total mass of the fractions should be within 1% of  $m_2$  or  $m_3$ , as appropriate. If it is not, then check for sieve damage and discard sieves as appropriate (see 8.2.1).

NOTE For practical purposes, it is usual to choose a standard sieve shaking time which gives an acceptable degree of sieving efficiency with a wide range of soil materials. The minimum recommended period is 10 min.

**Table 2 — Maximum mass of material to be retained on each test sieve at the completion of sieving**

Test sieve aperture mm	Maximum mass kg				
	Sieve diameter mm				
	450	400	300	200	100
50	10	8,9	4,5	—	—
37,5	8	7,1	3,5	—	—
28	6	5,3	2,5	—	—
20	4	3,6	2	—	—
14	3	2,7	1,5	—	—
10	2	1,8	1	—	—
6,3	1,5	1,3	0,75	—	—
5	1	0,9	0,5	—	—
3,35	—	—	—	0,3	0,15
2	—	—	—	0,2	0,1
1,18	—	—	—	0,1	0,05
0,6	—	—	—	0,075	0,0375

**Table 2** (continued)

Test sieve aperture	Maximum mass				
	kg				
	Sieve diameter				
mm	mm				
	450	400	300	200	100
0,425	—		—	0,075	0,0375
0,3	—		—	0,05	0,025
0,212	—		—	0,05	0,025
0,15	—		—	0,04	0,02
0,063	—		—	0,025	0,0125

**8.4 Calculation and expression of results**

For the material retained by the 20 mm and larger aperture sieves, calculate the proportion by mass retained by each sieve as a proportion of  $m_1$ . For example (Formula (2)):

$$\text{Proportion retained on the 20 mm sieve} = [m(20\text{mm})] / m_1 \tag{2}$$

For the material passing the 20 mm sieve, multiply the mass of material passing each sieve by  $m_2/m_3$  and calculate this as a proportion of  $m_1$ . For example (Formula (3)):

$$\text{Proportion retained on the 6,3mm sieve} = m(6,3\text{mm}) [(m_2/m_3) / m_1] \tag{3}$$

Present the results as a Table showing, to two significant figures, the proportion by mass retained on each sieve and the proportion passing the 2 mm sieve. The data shall also be used to construct a cumulative distribution curve (see Figure 1).

**9 Wet sieving and sedimentation (material <2 mm)**

**9.1 General**

This clause specifies the procedure (see Figure 2) for the determination of the particle size distribution of the material passing the 2 mm aperture sieve down to <0,002 mm equivalent spherical diameter (see note). In order to ensure that primary particles, rather than loosely bonded aggregates, are measured, organic matter and salts are removed, especially sparingly soluble salts such as gypsum which would otherwise prevent dispersion and/or promote flocculation of the finer soil particles in suspension (see 9.6), and a dispersing agent is added (9.3.2). These procedures are required in this document, and their omission shall invalidate its application. Sometimes iron oxides and carbonates, especially of calcium and/or magnesium, are also removed. Preferred procedures for the removal of these compounds are given in the note in 9.7. The removal of any compound shall be recorded in the test report (see Clause 10).

NOTE 1 Gravitational sedimentation can give a value for the total amount of material <0,002 mm equivalent spherical diameter. However, the method cannot be used to divide this class further with reliability, as particles less than about 0,001 mm equivalent spherical diameter can be kept in suspension almost indefinitely by Brownian motion (see Reference [2]).

**9.2 Apparatus**

The apparatus specified hereafter is sufficient to deal with one sample. Clearly it is more efficient to work in batches. Experience has shown (see Reference [7]) that one operator can process up to 36 samples in a batch at a time, given sufficient apparatus and space, especially if calculations are dealt with by a computer.

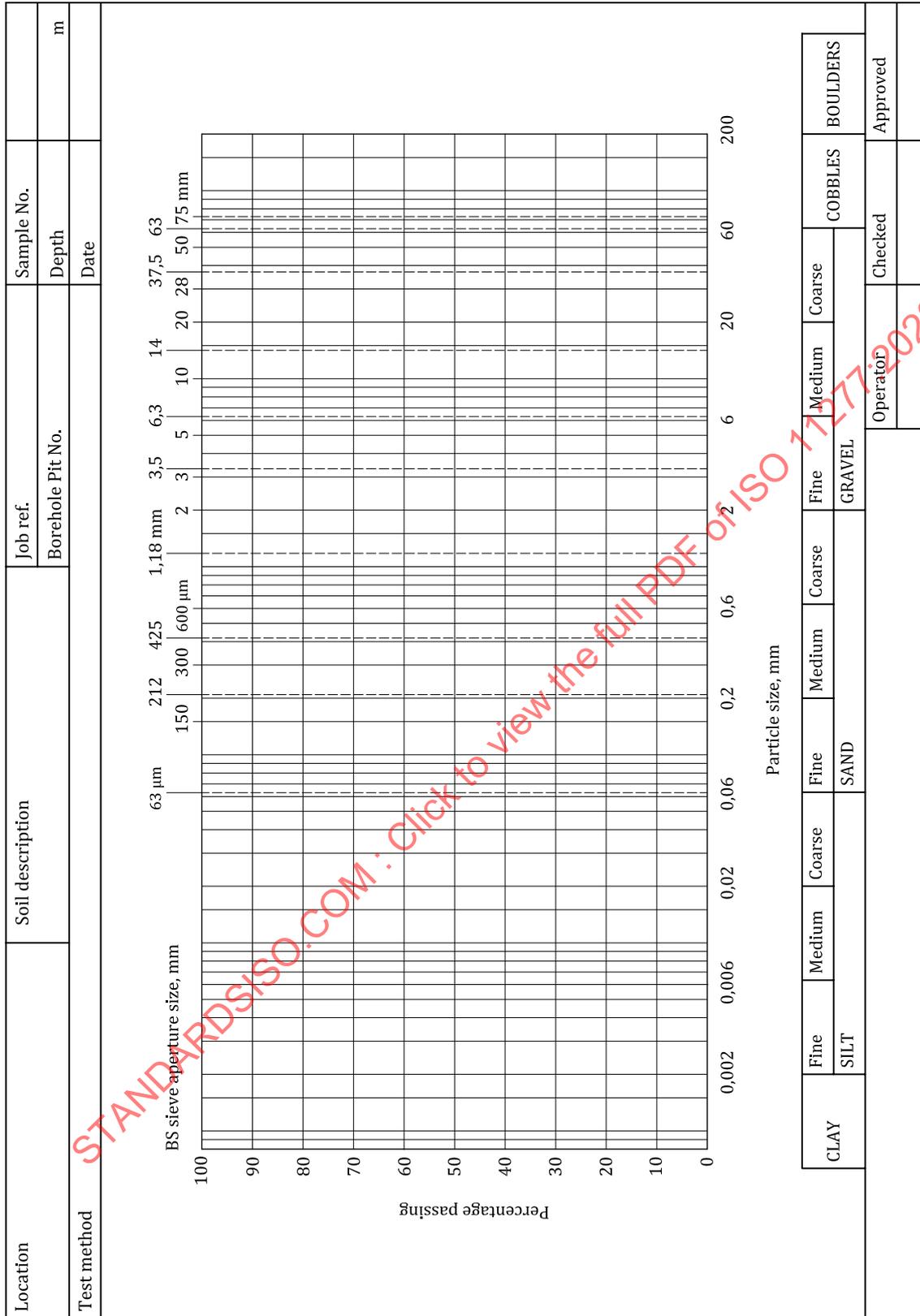
**9.2.1 Sampling pipette or sampling needle**, of a pattern similar to that shown in [Figure 3](#) with sideways openings, the chief requirement being that the smallest practicable horizontal zone of sedimenting suspension shall be sampled. The pipette shall be of not less than 10 ml volume and shall be held in a frame so that it can be lowered to a fixed depth within a sedimentation tube (see [Figure 4](#)).

NOTE 1 Experience suggests that a pipette with an upper volume of 50 ml is more than sufficient for most purposes. A 25 ml volume pipette is a convenient compromise for routine analysis, but a smaller volume pipette will be found to be sufficient for soils with down to about 10 % mass fraction of <0,063 mm equivalent spherical diameter. Below this amount, greater precision is likely to be obtained with a pipette of larger volume.

NOTE 2 The position of the pipette is adjusted to the new suspension surface after each sampling.

NOTE 3 Automated systems can be used, if the results are proven to be equivalent.

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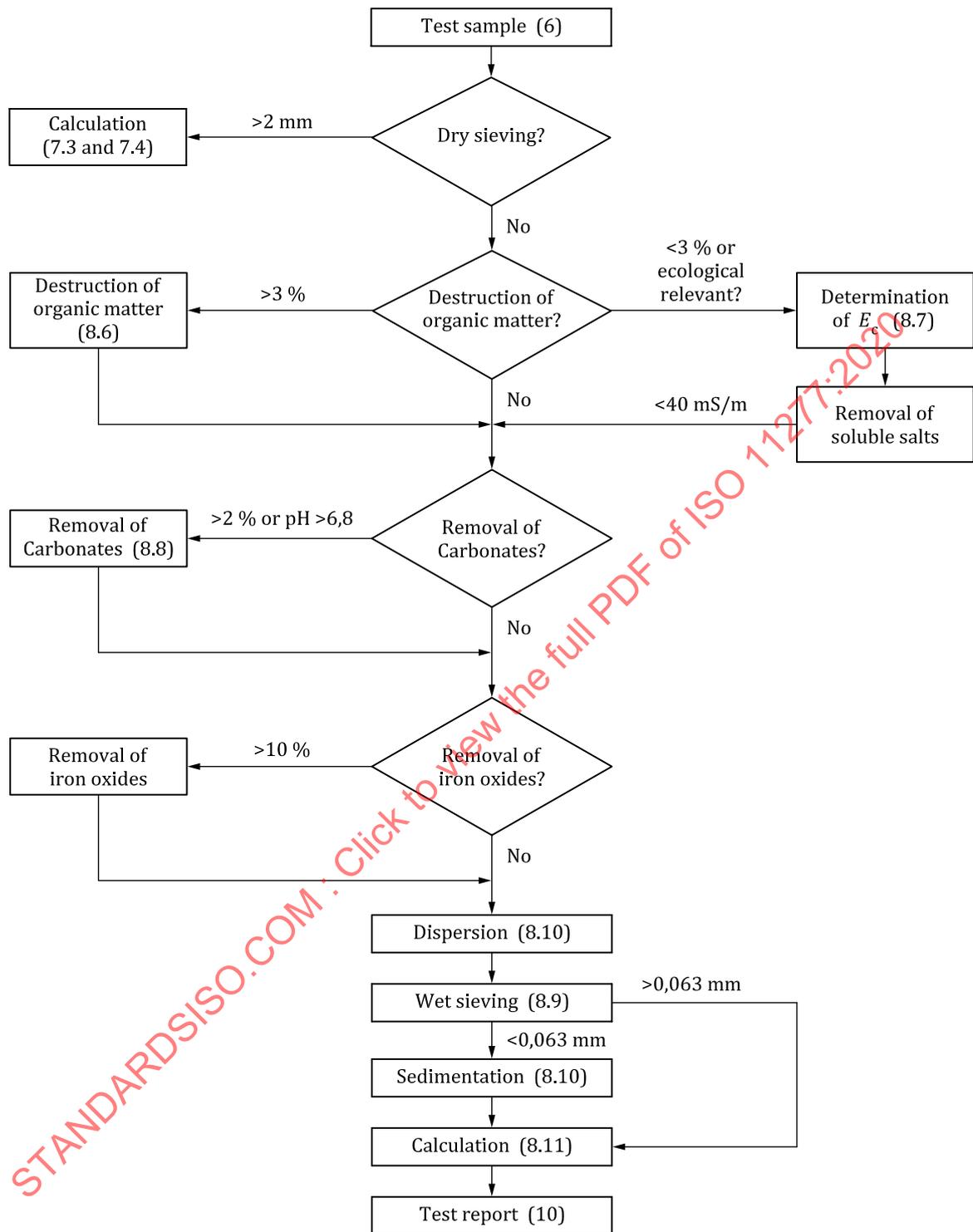
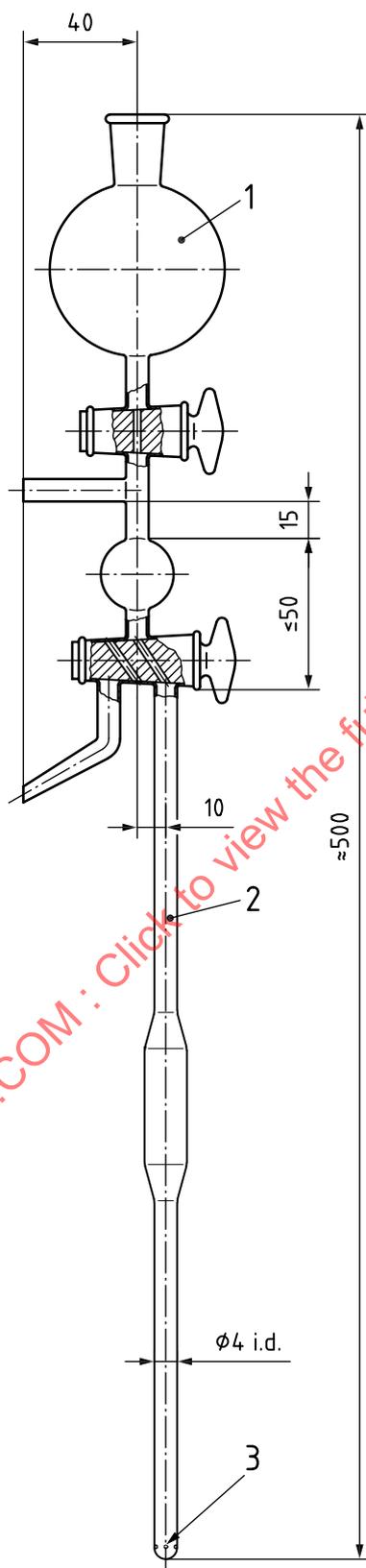


Figure 2 — Flow chart



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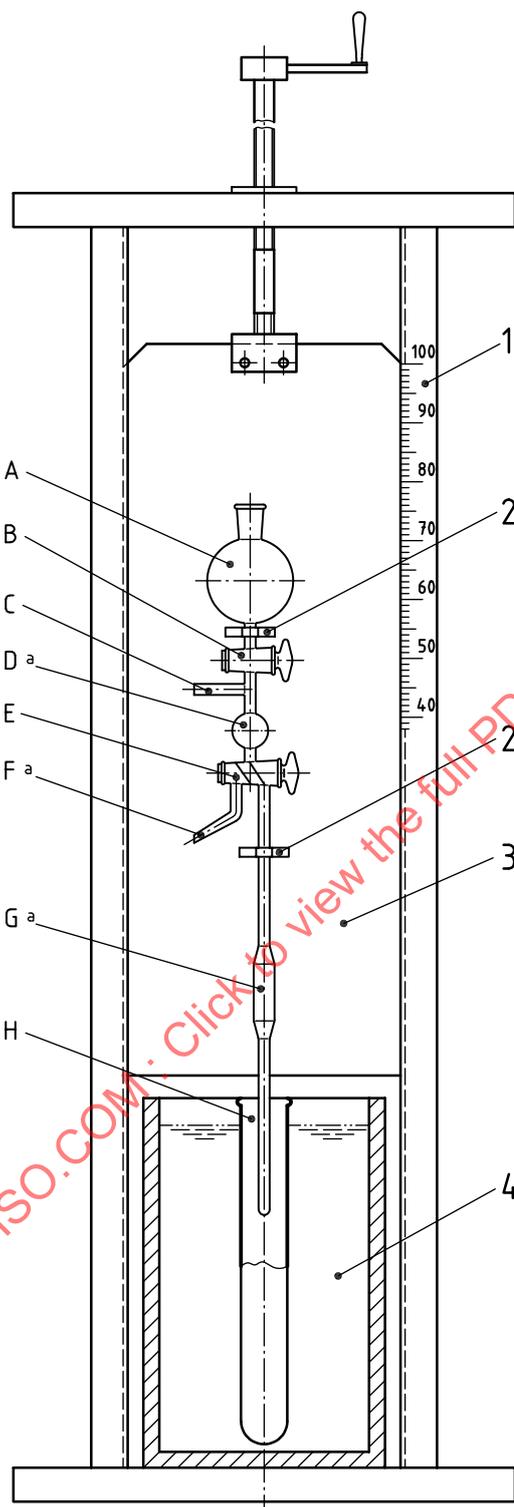
**Key**

- 1 bulb capacity: approximately 125 ml
- 2 pipette and changeover cock capacity at least 10 ml

NOTE This design has been found satisfactory, but alternative designs can be used.

**Figure 3 — Sampling pipette for sedimentation test**

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**Key**

- |         |                                  |   |                                |
|---------|----------------------------------|---|--------------------------------|
| A and B | 125 ml bulb funnel with stopcock | H | sedimentation tube             |
| C       | safety-bulb suction inlet tube   | 1 | scale graduated in millimetres |
| D       | safety bulb                      | 2 | clamps                         |
| E       | tap                              | 3 | sliding panel                  |

- F outlet tube 4 constant-temperature bath  
 G sampling pipette  
 a D, F and G are joined to three-way stopcock E.

NOTE This design has been found satisfactory, but alternative designs can be used.

**Figure 4 — Arrangement for lowering sampling pipette into soil suspension**

**9.2.2 Constant-temperature room or bath**, which can be maintained at between 20 °C and (30 ± 1) °C. If a bath is used, it shall accept a sedimentation tube immersed to the 500 ml or 1 000 ml mark and shall not vibrate the contents of the tube. Similarly, if a room is used, it, and its furniture, shall be constructed so that activity does not cause the tubes and their contents to vibrate.

NOTE This temperature range has been chosen to allow for the difficulties of maintaining one specified temperature in different parts of the world. In addition, the lower temperature gives sedimentation times that fit well into an average working day, whilst the upper temperature still allows for a sensible settling time for the fraction 0,063 mm equivalent spherical diameter (see [Clause 4](#) and [Table 3](#)).

**Table 3 — Pipette sampling times and  $d_p$  (for a particle density of 2,65 Mg/m<sup>3</sup>) at a sampling depth from the surface of (100 ± 1) mm at different temperatures assuming a particle density of 2,65 Mg/m<sup>3</sup> and density of water with dispersant of 1,00 Mg/m<sup>3</sup>**

T	$\eta$	Times, after mixing, of starting sampling operation								
		1 <sup>st</sup> sample <sup>a</sup>		2 <sup>nd</sup> sample		3 <sup>rd</sup> sample		4 <sup>th</sup> sample		
°C	mPa/s	min	s	min	s	min	s	h	min	s
15	1,139	1	4	5	17	53	11	8	47	45
16	1,109	1	2	5	8	51	47	8	33	51
17	1,081	1	1	5	1	50	29	8	20	53
18	1,053	0	59	4	53	49	10	8	7	54
19	1,027	0	58	4	46	47	57	7	55	52
20	1,002	0	56	4	38	46	46	7	44	5
21	0,978	0	55	4	32	45	39	7	32	55
22	0,955	0	53	4	25	44	34	7	22	16
23	0,933	0	52	4	19	43	33	7	12	4
24	0,911	0	51	4	13	42	32	7	1	58
25	0,891	0	50	4	8	41	35	6	52	37
26	0,871	0	49	4	2	40	39	6	43	21
27	0,851	0	48	3	56	39	43	6	34	5
28	0,833	0	47	3	51	38	53	6	25	44
29	0,815	0	46	3	46	38	2	6	17	24
30	0,798	0	45	3	42	37	14	6	9	31
31	1,781	0	44	3	37	36	27	6	1	39
32	0,764	0	43	3	33	35	42	5	54	11
33	0,749	0	42	3	28	34	58	5	46	57
34	0,734	0	41	3	24	34	16	5	39	58
35	0,719	0	40	3	20	33	35	5	33	12
$d_p$ (mm)		0,063		0,02		0,0063		0,002		
Sampling depth (m)		0,2	0,2	0,1	0,1	0,1	0,1	0,1	0,1	0,1

<sup>a</sup> Sampling depth (200 ± 1) mm to allow adequate time for the stabilization of the suspension after mixing.

**9.2.3 Glass sedimentation tubes**, without pouring lips, of internal diameter approximately 50 - 60 mm, and overall length of approximately 350 mm – 450 mm, graduated at 500 ml or 1 000 ml volume, and with either rubber bungs to fit or a stirrer.

**9.2.4 Stirrer**, of non-corrodible material.

**9.2.5 Weighing vessels**, inert material, with masses known to the nearest 0,000 1 g.

**9.2.6 Mechanical shaker**, capable of keeping 30 g of soil in suspension in 150 ml of liquid.

NOTE For keeping the soil in suspension, a suitable device could be an end-to-end type of shaker (e.g. 10 to 30 revolutions/min) or a stirrer.

**9.2.7 Test sieves**, complying with ISO 565, ISO 3310-1 and ISO 3310-2, having apertures of 2 mm and 0,063 mm, plus two intermediate sieves. The test report shall state which apertures are used. Round-hole sieves shall not be used.

NOTE The choice of the sieve of aperture 0,063 mm given here is for illustration but accords with the widespread use of this particle size to define the upper boundary of the silt fraction. Local requirements can specify another aperture. The choice of apertures for the intermediate sieves is a matter for local knowledge, but experience suggests that sieves of aperture close to 0,2 mm and 0,1 mm are useful for a very wide range of soils.

**9.2.8 Suitable sample divider** (see [Clause 6](#)).

**9.2.9 Balance**, capable of weighing to an accuracy of within  $\pm 0,000$  1 g.

**9.2.10 Drying oven**, capable of maintaining a temperature between 105 °C and 110 °C.

**9.2.11 Stop clock**, readable to 1 s.

**9.2.12 Desiccator**, containing anhydrous silica gel (preferably of the self-indicating type), capable of holding the five weighing vessels. The desiccant shall be inspected daily and dried at between 105 °C and 110 °C when it is no longer effective.

**9.2.13 Glass beaker**, of capacity 650 ml with a cover glass to fit, or a 300 to 500 ml **centrifuge bottle** with a leak proof cap.

NOTE This apparatus is used for chemical pre-treatment during which a constant problem is the adhesion of very fine particles to glass. The problem is much reduced if the treatment is carried out in a polycarbonate or polysulfone centrifuge bottle. Both materials will withstand repeated heating to 120 °C and are resistant to hydrogen peroxide and common dispersing agents. Their use can also save significant amounts of operator time.

**9.2.14 Centrifuge**, capable of holding the 300 to 500 ml centrifuge bottles (see [9.2.13](#)).

**9.2.15 Measuring cylinder**, of capacity 1 000 ml.

**9.2.16 Pipette**, of capacity between 10 to 50 ml (25 ml recommended) (see [8.2.1](#)).

**9.2.17 Glass filter funnel**, capable of holding the 0,063 mm sieve.

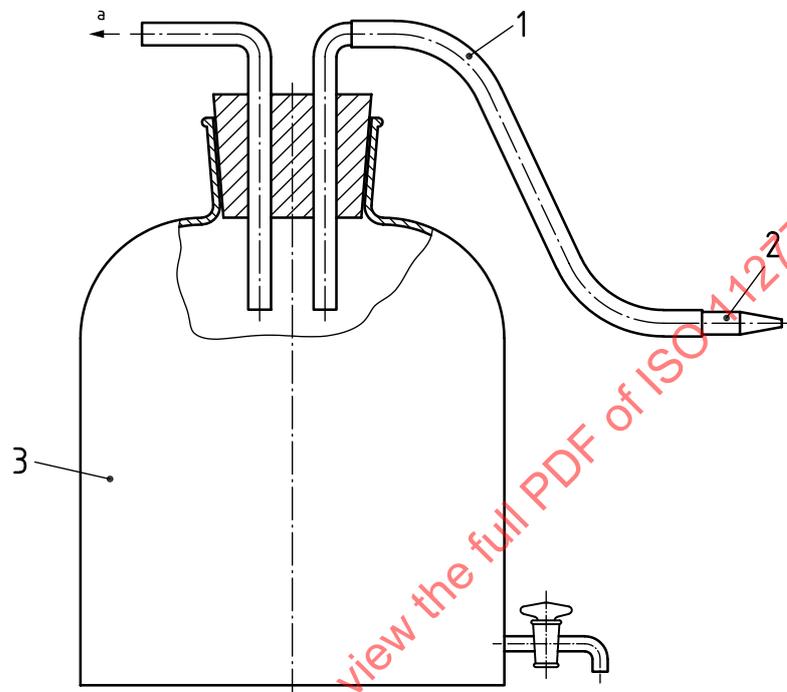
**9.2.18 Wash bottle** containing water (see [8.3](#)).

**9.2.19 Rod**, of glass or strong plastic, 150 mm to 200 mm long and at least 4 mm in diameter, with a rubber sleeve at one end.

**9.2.20 Electric hotplate**, capable of maintaining a temperature between 105 °C and 110 °C or **Water bath**, capable of maintaining a temperature between 90 °C and 95 °C.

NOTE A hotplate is essential if polymer centrifuge bottles are used for the chemical pretreatment, but a Bunsen burner, gauze and tripod are sufficient if glass beakers are used.

**9.2.21 Suction device**, similar to that shown in [Figure 5](#) is useful, but not essential.



**Key**

- 1 flexible tube
- 2 Pasteur pipette or similar
- 3 reservoir (5 l or 10 l)
- a To vacuum.

**Figure 5 — Sketch of suction device**

**9.2.22 Sieve brush.**

**9.2.23 Electrical conductivity meter**, accurate to 1 mS/m.

### 9.3 Reagents

All reagents shall be of recognized analytical grade. Use water having an electrical conductivity no greater than 10 mS/m at 25 °C at the time of use.

**9.3.1 Hydrogen peroxide solution, 30 % volume fraction.**

NOTE A 30 % volume fraction solution is one which will yield 30 ml of gaseous oxygen from 100 ml of solution (under standard conditions of temperature and pressure) upon reduction to water, either by chemical means or by boiling.

### 9.3.2 Solution of a dispersing agent.

As a dispersing agent, dissolve 33 g of sodium hexametaphosphate and 7 g of anhydrous sodium carbonate in water to make 1 l of solution. Store away from strong sunlight and preferably in a dark bottle. Record the date of preparation on the bottle. The solution is unstable and shall be replaced after one month.

Another suitable dispersant is 0,1 M sodium pyrophosphate solution (tetrasodiumdiphosphate decahydrate  $\text{Na}_4\text{P}_2\text{O}_7 \cdot 10 \text{H}_2\text{O}$ ).

The sodium carbonate buffers the solution and the suspension of the soil, to about pH 9,8. The described dispersing agents have been found successful with a very wide range of soils. However, if there are signs that dispersion is ineffective, consider firstly that flocculating salts might be present (see 9.7). If dispersion is still unsuccessful after removal of salts, then other dispersing agents should be considered. A very effective but less widely used dispersing agent is prepared by replacing the sodium carbonate with 20 % volume fraction ammonia solution, in the ratio of 5 ml ammonia solution to 150 ml of the hexametaphosphate solution. There are many other dispersing agents (see Reference [3]). Whichever is chosen, considerable investigation will be required to establish its effectiveness. It is noticed that some soils show fewer problems of dispersion if analysed without drying (see Annex A). Some soils derived from recent volcanic deposits will disperse more effectively in an acid medium (see Reference [10]).

### 9.3.3 Octan-2-ol, or a similar volatile antifoaming agent.

NOTE Octan-2-ol is highly effective and relatively long-lasting. Ethanol or methanol can also be used, but the use of pentan-2-ol (amyl alcohol) is discouraged because it is potentially addictive.

## 9.4 Calibrations

### 9.4.1 Sampling pipette (see Figure 4)

Clean and dry the pipette thoroughly and immerse the tip in water held at the same temperature as that of the constant-temperature environment (9.2.2). By means of a tube attached to C, draw water into the pipette above E. Drain off the water above E through F. Drain the pipette into a weighing vessel (9.2.5) of known mass and determine the new mass. From the known masses, calculate by use of the density the internal volume of the pipette. Repeat this exercise three times and take the average of the three volumes as the internal volume of the pipette to the nearest 0,05 ml ( $V_c$  ml).

### 9.4.2 Dispersing-agent correction

Follow this procedure each time a new batch of dispersing agent is prepared.

Pipette 25 ml of dispersing-agent solution into one of the glass sedimentation tubes, and fill the tube to the 500 ml mark with water. Mix the contents of the tube thoroughly. Place the tube in the constant-temperature environment, and leave the tube for at least 1 h. Between any of the times at which samples may be taken from the sampling tube (Table 3), take a sample ( $V_c$  ml) of the dispersing-agent solution from the sedimentation tube using the sampling pipette. Drain the pipette into a weighing vessel of known mass, and dry the contents of the vessel between 105 °C and 110 °C. Allow the vessel to cool in the desiccator and determine the mass of the residue in the vessel to 0,000 1 g ( $m_r$ ).

## 9.5 Test sample

The test sample shall be taken from the material passing a 2 mm aperture sieve (see Clause 7 and 9.2) and weighed to the nearest 0,001 g ( $m_s$ ). The mass of test sample depends on the type of soil. Approximately 30 g for a sandy soil and 10 g for a clay soil are appropriate for pipette analysis, with proportionate masses for soils intermediate to these extremes. For the hydrometer method (Annex B),

take twice this amount of material. Place the test sample in either the 650 ml glass beaker or the 300 to 500 ml centrifuge bottle (9.2.13 and its note).

NOTE 1 Soils rich in organic matter contain relatively little mineral matter. It might be necessary to take up to 100 g of such soils in order to obtain sufficient mineral matter for a reliable analysis of the particle size distribution of this component. Such a large amount of organic material is preferably apportioned between several vessels for ease of operation, with combination of the mineral residues at a later stage.

The dispersion is done by chemical and mechanical means. Chemical measures include the (optional) removal of the (micro) aggregates stabilizing substances, i.e. organic matter (9.6), the control and the establishment of salt content (9.7), iron and aluminium oxides (9.9) and carbonates (9.8), and the addition of dispersing agents (9.10) that are required for the peptisation of the clay particles and the stabilisation of the entire suspension.

Flocculation and dispersion are influenced by the salts in the soil solution by two effects which are not independent:

- 1) charge effect: When divalent and trivalent cations such as  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  and  $\text{Al}^{3+}$ , the charge density is so high that a strong electrostatic attraction between cation and clay mineral is concluded. Polyvalent cations act as bridges between the clay minerals and connect them to form aggregates or flocculation in the suspension. Monovalent cations are less able to; clays peptize (= disperse) under the influence of monovalent cations. Thus dominate monovalent cations such as Na, especially at the exchange sites of the clay minerals in the soil solution, and, results in weaker aggregation of the clay particles in the presence of polyvalent cations.
- 2) concentration effect: A high absolute salinity is independent of the type of ions generally stabilizing effect on the structure, since he thus causes flocculation of the clay particles aggregate into larger units. The higher the proportion of the sodium cation, the higher the total salt content shall be, in order to effect flocculation of the clay and thus a stable floor structure.

These effects are used for chemical dispersion by the addition of sodium hexametaphosphate or sodium pyrophosphate:

- due to the high concentration of sodium and polyvalent cations on the clay particles;
- transfer from the phosphate exchanged cations into insoluble complexes and thus prevent flocculation.

## 9.6 Destruction of organic matter

### 9.6.1 General

The removal of the organic substances is recommended if the organic matter content is >3%. For the determination of ecologically relevant particle size distribution the destruction of organic matter is not necessary (see NOTE).

NOTE Under field conditions, organic matter, calcite and iron oxides contribute to the particle size responsible for physical characteristics like pore space or water retention capacity. The destruction of these components can allow for a precise measurement of the mineral particle size, which might be different from the “ecologically relevant” particle size though.

Destroy the organic matter with hydrogen peroxide solution following method A or B described below.

Samples prone to very strong reaction should be treated with smaller portions or lower concentration (e.g. 10 % solution) of hydrogen peroxide solution (9.3.1).

**WARNING — Carry out this step with caution. Hydrogen peroxide can decompose violently with some forms of organic matter, manganese compounds and finely-particulate iron sulfides, all of which can occur in soil. Do not examine the reaction by looking into the top of the vessel. Do not accelerate an apparently slow initial reaction by heating or addition of more hydrogen peroxide.**

### 9.6.2 Method A

Fill the test sample either in a glass beaker or a centrifuge bottle (9.2.13), add approximately 30 ml of water to the test sample and allow it to become thoroughly wet. Add 30 ml hydrogen peroxide solution and mix the contents of the vessel very gently using the glass or plastic rod. A vigorous reaction can cause frothing of the sample mixture. This can be controlled by adding a few millilitres of octan-2-ol. Allow any vigorous reaction to subside. Cover with the cover glass and leave for at least 3 hours. Place the vessel on the hotplate or Bunsen burner, and warm gently. Control any frothing with octan-2-ol as before, and stir the contents frequently. Do not allow the contents to dry out, adding more water if necessary. Bring the suspension to a gentle boil and heat until all signs of bubbling due to decomposition of hydrogen peroxide have ceased. If there is still-undecomposed organic matter, remove the vessel from the heat, allow it to cool and repeat the treatment with hydrogen peroxide. Organic soils will need several such treatments; the products of the reaction being removed after every 2 or 3 treatments before continuing with more peroxide.

Transfer the contents to a centrifuge bottle, taking care to remove all traces of material from the sides of the beaker by means of the rubber sleeve on the glass or plastic rod. The final volume should be 150 ml to 200 ml.

### 9.6.3 Method B

Fill the test sample either in a glass beaker or a centrifuge bottle (9.2.13), add water to the test sample and allow it to become thoroughly wet. Place it in an ultrasonic heating bath at  $(70 \pm 5) ^\circ\text{C}$  and add in small portions in total 75 ml hydrogen peroxide solution within a period of 3 h. The addition of hydroxide peroxide shall be done in small portions, with care, in order to prevent violent reactions. In case of a strong reaction of the sample, a little water or octan-2-ol can be added.

Add additional portions of 25 ml hydrogen peroxide to digest all remaining organic matter. After the addition of a portion hydrogen peroxide stir or apply low ultrasonic power to the bath for 1 minute. After the last addition of peroxide let the reaction subside and apply high ultrasonic power for 1 min at  $(70 \pm 5) ^\circ\text{C}$ .

NOTE 1 Lignified (woody) residues of plants are extremely difficult to decompose, and their complete destruction is often impossible. Such fragments are usually regarded as decomposed when they have lost all traces of dark colour.

Transfer, in case a beaker was used, the washed residue quantitatively to a centrifuge bottle. In all cases, it is not essential for the supernatant to be absolutely colourless, so long as it is obvious that the bulk of the dark decomposition products of the organic matter have been removed, but the solution shall be clear.

The removal or not removal of organic matter shall be recorded in the test report. If organic matter was removed, even the use of calcium chloride solution or aluminium sulfate solution, in conjunction with filtration, decantation, suction or siphoning, shall be recorded in the test report (see [Clause 10](#)).

Continue with the removal of carbonate (if needed) or salts and fill up to about 200 ml of water and centrifuge the container.

NOTE 2 Dry organic materials are often strongly hydrophobic, in which case the addition of a few drops of octan-2-ol can be beneficial.

**WARNING — Ensure that there is no more hydrogen peroxide in the sample, otherwise the shake flasks can burst during the subsequent shaking due to the arising pressure.**

Centrifuge the bottle to obtain a clear supernatant [15 min at a minimum relative centrifugal force (RCF) of 400g is recommended] and decant the latter or remove by means of the suction device. Repeat the treatment until the supernatant is colourless or nearly so.

If a centrifuge is not available, the mineral residues may be flocculated by adding 25 ml of 1 mol/l calcium chloride solution. Stir thoroughly, bring to about 250 ml with water, allow standing until the

supernatant is clear, then siphon or decant this from the residue. Add another 250 ml of water and repeat the washing procedure until the dark residues of the decomposed organic matter have gone.

Check that the electrical conductivity of the washings is below 100 mS/m before attempting to disperse the residue (see 9.7).

Another alternative is to filter the residue from the oxidation step on a hardened, high wet-strength filter paper (2,7  $\mu\text{m}$  pore size is suitable), followed by thorough washing with water by means of the wash-bottle. It is essential to observe the filtrate closely to see that no soil is lost. If particles pass through the filter paper, return the filtrate to the container, add calcium chloride solution to the suspension as above, stir and refilter.

If flocculation with calcium chloride, or filtration, are ineffective in preventing the loss of fine soil particles, then a few drops of a 60 g/l aluminium sulfate solution can be stirred into the soil suspension. The absolute minimum of aluminium sulfate shall be used, as excess could cause problems in the subsequent dispersion of the soil.

### 9.7 Removal of soluble salts and gypsum

Soluble salts and gypsum shall be removed if the electrical conductivity  $\gamma$  is above 40 mS/m in accordance with ISO 11265, because they might interfere with dispersion. If also the optional removal of the carbonates will be done, then both steps are performed together during the removal of carbonates.

NOTE Soils having electric conductivities higher than 40 mS/m (400  $\mu\text{S}/\text{cm}$ ) in the supernatant, most often will flocculate in an aqueous suspension due to their natural concentration of salt.

Add sufficient water to the soil in the centrifuge bottle (9.2.13) so that the soil:water ratio is between 1:4 and 1:6 by volume. Shake the contents of the bottle vigorously so that all the sediment is in suspension; then shake for 1 h on the end-over-end shaking machine. Centrifuge to obtain a clear supernatant and measure its electrical conductivity. If the latter is  $<40$  mS/m, soluble salts and gypsum are not present in significant amounts. If the value of  $\gamma > 40$  mS/m, then remove the supernatant in which  $\gamma$  was measured. Add 250 ml of water to the soil residue, cap the bottle and shake on the end-over-end shaking machine for 1 h. Centrifuge to obtain a clear supernatant and measure  $\gamma$  again. If it is  $<40$  mS/m, soluble salts and gypsum have been removed to an extent sufficient not to interfere in the dispersion (9.10). If the  $\gamma$  is  $>40$  mS/m, repeat the washing procedure until the  $\gamma$  of the supernatant is  $<40$  mS/m.

The removal of soluble salt or gypsum shall be recorded in the test report (see Clause 10).

### 9.8 Removal of carbonates

The removal of the carbonate is optional; it can be carried out together with the removal of the soluble salts and the gypsum.

Calcium and magnesium carbonates are removed by treating the soil with the minimum possible excess of aqueous hydrochloric acid.

The following procedure has been found to be applicable to a wide range of soils and is best applied to the soil after the removal of organic matter (9.6). Where the mass fraction of carbonate is greater than about 2 %, add, to the washed, centrifuged soil (see above), 4 ml of 1 mol/l hydrochloric acid for each percent of carbonate, plus an excess of 25 ml of acid. Make up to about 250 ml with water and place the suspension on the water bath at about 70 °C to 80 °C for 15 min, stirring the suspension from time to time. Remove from the water bath and leave the suspension to stand overnight. If the soil flocculates sufficiently to leave a clear supernatant, then this can be siphoned off or decanted, otherwise centrifugation (see 9.6) and decantation will be necessary. Repeat the washing and decantation with water until the  $\gamma_c$  of the supernatant is less than 100 mS/m. If the mass fraction of carbonate is less than about 2 %, then only an initial 25 ml of 1 mol/l hydrochloric acid solution is required. However, there is then the risk that there will be insufficient calcium in solution to give good flocculation. It is recommended, therefore, that 20 ml of 1 mol/l calcium chloride solution be added at the same time as the acid. The rest of the procedure is identical for both situations.

If magnesium carbonates are present in substantial amounts, then the treatment can be lengthy.

Hot hydrochloric acid shall not be used in soils containing significant amounts of chlorite, as this can dissolve.

The removal of carbonates shall be recorded in the test report (see [Clause 10](#)).

NOTE 1 Because of the slowness of the procedure and the difficulty of quantitative recovery of the residue, removal of carbonates is not normally a routine procedure in the determination of particle size distribution.

NOTE 2 If the removal of the carbonate is generally intended, however, uncertainty about the carbonate content of the sample exists, it can be decided on the basis of the pH (H<sub>2</sub>O). At pH values above 6.8 carbonate is assumed to be present in significant amounts; samples with pH values below 6.8 can be dispensed from the removal of the carbonate.

### 9.9 Removal of iron oxides

The removal of the iron oxides (and associated aluminium) is optional for samples with contents >10 %. For samples with contents <10 %, no removal is necessary.

Iron oxides are removed by shaking the soil overnight with 40 g/l sodium dithionite in approximately 0,3 mol/l sodium acetate solution buffered to pH 3,8 with acetic acid, in the ratio of 1 part soil to 40 parts of solution, both by volume.

NOTE Very iron-rich soils usually need several treatments. Magnetite is not affected by this procedure<sup>[5]</sup>. In certain soils, especially those developed from recent volcanic deposits, this reagent can remove large quantities of aluminium as well as iron.

Afterwards, the sample is centrifuged and the clear supernatant can be decanted or siphoned off until the  $\gamma$  is <100 mS/m.

The removal of iron/aluminium oxides shall be recorded in the test report ([Clause 10](#)).

### 9.10 Dispersion

Add sufficient water to the centrifuge bottle so that the total volume is between 150 ml and 200 ml. Add 25,0 ml of dispersion agent with a pipet or dispenser. Then select one of the three procedures (A, B or C) In case of ecological particle size determination option A is the preferred:

- a) Shake the bottle for overnight on the end-over-end shaker at 6 rpm. Ensure that, if soils are shaken over the weekend, the total shaking time does not exceed 18 h;
- b) Increase the temperature of the solution to boiling temperature and keep it for (5 ± 0,5) min. Cool the solution to room temperature;
- c) Apply high ultrasonic power in the waterbath for 10 min. at (70 ± 5) °C.

If an end-over-end shaker is not available, then a vibrating blade stirrer can be used, unless destructible aggregates are present. But the blender type of stirrer should not be used because it can comminute primary particle.

### 9.11 Wet sieving at 0,063 mm

Place a 0,063 mm aperture sieve in the large glass funnel and place the funnel in the stand so that the neck of the funnel is inside one of the 500 ml sedimentation tubes. Transfer the dispersed suspension from the centrifuge bottle quantitatively onto the sieve, and wash the soil using a jet of water from the wash-bottle until the water runs clear. If necessary, agitate the suspension on the sieve to alleviate

sieve blockage, using the glass or plastic rod and rubber sleeve. Take great care to avoid damage to the sieve mesh when this is done. The total volume of the washings should not exceed 500 ml.

NOTE Certain highly weathered tropical soils can be difficult to sieve, and it can help to wet the sieve by gently rubbing a few drops of dispersing agent over and under the mesh prior to this step.

Remove the sieve from the funnel and wash the residue on the sieve into an evaporating dish by means of a gentle spray from the wash-bottle. Place this dish in an oven between 105 °C and 110 °C until the residue is dry. Wash any particles adhering to the inside of the funnel into the sedimentation tube. Allow the dried residue to cool, and re-sieve on the sieves <2 mm down to 0,063 mm, returning any material passing the latter to the sedimentation cylinder. Weigh the fractions retained on each sieve, and record their masses. Make up the suspension in the sedimentation tube to 500 ml with water.

## 9.12 Sedimentation

Place the sedimentation tube in the constant-temperature environment. If a bath is used, the tube shall be immersed to the 500 ml mark. Allow the tube and contents to equilibrate with the temperature of the system (see the last paragraph of 9.4.2). The change in the temperature of the suspension should be less than  $\pm 1,5$  °C during the settling period.

Agitate the contents of the sedimentation tube vigorously, either by means of the stirrer, which should be used with a powerful plunging action, or by inserting a bung in the tube, followed by end-over-end shaking. In both cases, no material shall be left adhering to the base of the tube. The contents of the tube shall be shaken or stirred at least 10 times per minute. Replace the tube upright in the constant-temperature environment (within  $\pm 1,5$  °C during sedimentation).

Long living vortices in the sedimentation tube that impede sedimentation shall be avoided.

The instant the tube and contents are placed upright or stirring with the plunger stops, start the timer. If rubber bungs have been used, then remove them from the cylinder, taking care not to agitate the cylinder whilst doing so. About 15 s before a sample is to be taken (see Table 3), lower the pipette, with tap E (see Figure 4) closed, vertically into the soil suspension and centrally in the sedimentation tube, until the tip is the appropriate depth ( $\pm 1$  mm) below the suspension surface (see Table 3). Take care to disturb the suspension as little as possible, and complete the operation within about 10 s. Open tap E and withdraw a sample of the suspension such that the pipette and the bore of tap E are full. This sampling operation shall take about 10 s. Withdraw the pipette from the suspension so that the tip of the pipette is clear of the top of the sedimentation tube.

During the sampling, some of the suspension can be drawn above tap E into bulb D (see Figure 4). Run this surplus into a small beaker down outlet tube F by opening tap E so that E and D are connected. Wash with water from reservoir A into D and out through F until no suspension remains in this part of the system.

Place a weighing vessel of known mass, to the nearest 0,000 1 g, under the tip of the pipette and open tap E so that the contents of the pipette are delivered to the vessel. Wash any suspension left on the inner walls of the pipette into the vessel by allowing water from reservoir A to run through the system. Place the weighing vessel and its contents in the oven between 105 °C and 110 °C and evaporate to dryness. Cool the vessel in the desiccator, weigh the vessel and its contents to the nearest 0,000 1 g, and determine the mass of the residue to the nearest 0,000 1 g ( $m_{s1}$ ). Clean the outside of the pipette of any adhering sediment, and take further samples, as required, in accordance with the times given in Table 3, using the same pipetting procedure as given above. Call the additional sample masses  $m_{s2}$ ,  $m_{s3}$ , etc. The fractions 0,063 mm to 0,002 mm and <0,002 mm shall be determined as a minimum. The construction of a particle-size-distribution curve requires that at least two other fractions intermediate between these two points be determined.

NOTE The choice of the two fractions is a matter for local decision. Experience suggests that it is more important to determine the amount of material below 0,032 mm equivalent spherical diameter than material above that size, if a reliable particle-size-distribution curve is to be drawn.

**9.13 Calculation of results for fractions <2 mm**

The method of calculation assumes that the sample mass is the sum of the constituent fractions, and not the mass of sample taken as specified in 9.5.

NOTE This approach has its drawbacks in that it assumes that there are no significant errors at various stages of the sampling, drying and weighing of each fraction. However, in order that this document be applicable to the widest range of soils, it is believed that this assumption is preferable to the risk of errors arising from the inadequate dispersion of oven-dried soil. The method given in this sub clause is thus the preferred method.

Calculate the mass of solid in suspension in 500 ml ( $mf_1, mf_2, mf_3$ , etc.) in grams, for each pipette sampling time from the [Formula \(4\)](#):

$$mf_x = ms_x (500/V_c) \tag{4}$$

where

$mf_x$  is the mass of solid in suspension in 500 ml, in grams;

$ms_x$  is the mass of material from the xth pipette sampling, in grams;

$V_c$  is the calibrated volume of the pipette, in millilitres.

Similarly, the mass of solid material in 500 ml of dispersant solution,  $m_d$ , in grams, is given by [Formula \(5\)](#):

$$m_d = m_r (500/V_c) \tag{5}$$

where

$m_r$  is the mass of residue, in grams;

$V_c$  is the calibrated volume of the pipette, in millilitres.

By way of example, denote the masses of the pipetted fractions (calculated as in 500 ml) as follows:

- fraction <0,063 mm =  $mf_1$ ;
- fraction <0,020 mm =  $mf_2$ ;
- fraction <0,002 mm =  $mf_3$ .

The samples taken at the shorter time intervals will contain the material of smaller particle size and each sample will contain the same mass of dispersant, i.e.  $m_r$ . Thus the mass of the fractions, in grams, can be calculated as:

- mass of fraction <0,063 mm to <0,020 mm =  $mf_1 - mf_2 = m(0,063 \text{ mm to } 0,020 \text{ mm})$ ;
- mass of fraction <0,020 mm to >0,002 mm =  $mf_2 - mf_3 = m(0,020 \text{ mm to } 0,002 \text{ mm})$ ;
- mass of fraction < ,002 mm =  $mf_3 - m_d = m(< 0,002 \text{ mm})$ .

The principle is extended to other fractions sampled. The mass of sample <2 mm is thus the sum of the masses of the fractions obtained by wet sieving at 0,063 mm and the masses of the fractions obtained by calculation. Denote this total sample mass as  $m_v$  in grams.

Calculate the proportion in each fraction <2 mm as given by [Formula \(6\)](#):

$$\text{Proportion} = \text{mass of fraction} / m_t \quad (6)$$

If required, the proportions of material <2 mm in diameter can be recalculated as proportions of the total soil mass inclusive of material >2 mm in diameter, as given by [Formula \(7\)](#):

$$\text{Proportion in total soil mass} = P_t (<2 \text{ mm}) / P_s (<2 \text{ mm}) \quad (7)$$

where

$P_t (<2 \text{ mm})$  is the proportion of the fraction <2 mm;

$P_s (<2 \text{ mm})$  is the proportion of material <2 mm in the total soil ([Clause 7](#)).

Present the results as a Table showing the proportion in each size fraction to two significant figures. State clearly the basis on which the results are presented, i.e. as a proportion of the material <2 mm, or as a proportion of the total soil. In addition, the results shall also be presented as a cumulative particle-size-distribution curve ([Figure 1](#)).

Data regarding the precision of the method are provided in [Annex C](#).

## 10 Test report

The test report shall include the following information:

- a) a reference to this document, i.e. ISO 11277:2020;
- b) the date of the laboratory analysis;
- c) the methods of test used, specifying the parts of the standard used where alternatives exist, in accordance with the instructions given within this document;
- d) the results of the sieve and pipette analyses, or the hydrometer analyses, as appropriate;
- e) sufficient information to identify the sample unequivocally within the laboratory records.

## Annex A (normative)

# Determination of particle size distribution of mineral soil material that is not dried prior to analysis

### A.1 Introduction

This annex specifies the procedure for soils which should not be dried because of the resulting change in properties, especially their dispersion characteristics. Some loss of precision can be expected with this method, especially with cohesive soils, due to the greater difficulty in obtaining a representative sample, especially of the <2 mm soil. However, this loss of precision is likely to be less than the errors arising from analysis of the dried soil. The decision as to which soils should or should not be analysed from the wet state is a matter for local judgement. The soils most likely to need this approach are those developed from recent volcanic materials, certain highly oxidized tropical soils, and highly cohesive soils. The method involves the wet sieving of material down to 2 mm, below which size the material is discarded. A separate sample is taken for the determination of particles <2 mm in diameter. It is important that the two test samples be taken at about the same time from the field sample, so that they are submitted to the test procedure at similar gravimetric moisture contents. Samples of mass equal to the two test samples are taken for the determination of the dry mass of the material, and these masses are used in the calculation of the proportions of the size fractions present in the soil.

### A.2 Procedure for material retained on a 2 mm aperture sieve

#### A.2.1 Apparatus

**A.2.1.1 Set of test sieves**, in accordance with [8.2.1](#).

**A.2.1.2 Balance**, capable of weighing to within an accuracy of  $\pm 0,5$  g.

**A.2.1.3 Drying oven**, capable of maintaining a temperature between 105 °C and 110 °C.

**A.2.1.4 Several corrosion-resistant trays.**

Those made from aluminium alloy sheet about 2 mm thick, with sides about 25 mm deep and a base of length about 300 mm, will be suitable.

**A.2.1.5 Plastic bucket**, or other corrosion-resistant container of about 10 l capacity.

**A.2.1.6 Sieve brushes** and a **stiff brush** such as a wire brush.

**A.2.1.7 Sodium hexametaphosphate solution** or other suitable dispersing agent ([9.3.2](#)).

**A.2.1.8 Means of supporting the test sieves** and a **device for washing** the material on the sieves.

A length of 6 mm bore tubing attached to a tap, or a fine-jet watering-can will suffice for washing.

### A.3 The test sample

Take a mass of sample (Clause 6) for sieving in proportion to the largest particles present (see Table 1). Weigh the sample to the nearest 0,5 g and record the mass ( $m_1$ ). Take an equal mass of sample by an exactly equivalent procedure and record the mass. Place this sample in a non-corroding tray and dry it in an oven at between 105 °C and 110 °C. Record the dry mass to the nearest 0,5 g ( $m_2$ ).

### A.4 Dispersion

Place the undried test sample in the plastic bucket, add about 5 L of water and 350 ml of dispersing agent. The sample shall be covered by the solution. Mix the contents of the container gently and allow standing overnight.

NOTE It is impracticable to attempt to destroy organic matter in samples of this size. Note also that the remarks concerning dispersion, other than those related to problems of drying, given in the last paragraph of 9.3.2 could still apply.

### A.5 Sieving at 2 mm

Mix the sample in the bucket gently again, and then pour the suspension slowly onto a nest of sieves (see Clause 7). Table 2 gives the mass of sample to be taken in relation to sieve size. Several sievings of portions of the suspension might be needed. Make sure there is no material remaining in the bucket, and then gently brush the material on the largest-aperture sieve with the stiff brush to remove any material adhering to the primary particles. Ensure that no fragments of the latter are dislodged during this process. Wash the material on the sieves thoroughly and wash onto the sieve any material adhering to the brush; tap water will suffice for this purpose. Allow the material passing the 2 mm sieve to run to waste. Sieve the material on the nest of sieves, brush each fraction into a weighed dish and dry at between 105 °C and 110 °C. Record the mass retained on each sieve to the nearest 0,5 g. The material retained on the 2 mm sieve should be washed thoroughly, any further material passing the sieve being allowed to run to waste.

The extent to which coarse particles absorb water is impossible to estimate by eye. Unless local experience suggests strongly to the contrary, all fractions should be dried before weighing. It is realized that there is an apparent paradox in drying fractions separated by a wet process, and reporting dry-mass proportions. However, wet separation is practiced to avoid difficulties in dispersion of dried soil and to avoid changes in particle size distribution due to shrinkage and adhesion of particles upon drying. The method given is the preferred method. The masses of the fractions can be reported as proportions of each in the wet soil, if this is desired for some reason. The moisture content of each fraction and of the original material shall then be given in the test report. The practical difficulties of this approach in routine analysis are very real, and intercomparison of results between this and the preferred method will be difficult.

Transfer the material retained on the 2 mm aperture sieve to a non-corroding tray, and dry the contents in the oven at a temperature between 105 °C and 110 °C. Sieve this dried material on a 2 mm aperture sieve, and record the mass of the material retained, to the nearest 0,5 g. Reject any material passing this sieve.

### A.6 Calculation and expression of results

#### A.6.1 Calculation

Calculate the mass of material retained on each sieve as a proportion of the oven-dried sample mass  $m_2$ . For example:

Proportion retained on the 16 mm sieve =  $m(16 \text{ mm})/m_2$

### A.6.2 Expression of results

Present the results as a Table showing the proportion by mass, to two significant figures, retained on each sieve. In addition, the results shall be shown as a cumulative distribution curve (see [Figure 1](#)).

### A.7 Wet sieving of material <2 mm diameter

Sieve the field-moist sample on a 2 mm aperture sieve so as to obtain about 300 g of <2 mm diameter material. It is usually necessary to rub the sample gently over the sieve mesh to do this. Take care not to damage the sieve mesh or disaggregate primary particles. Take two subsamples, of about 30 g each, by quartering the sieved material. The masses of the two samples shall agree to within 0,5 g ( $m_3$  and  $m_4$ ).

NOTE It is convenient to keep a separate 2 mm sieve for this purpose. It can also be convenient to keep a complete set of sieves for wet sieving if this is to be carried out often. Place one subsample in a non-corroding tray and dry it as in [A.3](#). Record the mass of the dried sample ( $m_5$ ). The other subsample is taken for sieving and sedimentation in accordance with the procedures in [9.11](#) and [9.12](#), respectively.

### A.8 Calculation and expression of results

The proportions of material in the fractions <2 mm equivalent spherical diameter are calculated and expressed as in [9.13](#) as proportions of the oven-dried mass ( $m_5$ ).

### A.9 Test report

This shall be made in accordance with the criteria given in [Clause 10](#). In addition, the report shall state clearly that the analysis was made on undried material, and the relevant clauses of this document shall be stated.

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## Annex B (normative)

### Determination of particle size distribution of mineral soils by a hydrometer method following destruction of organic matter

#### B.1 Introduction

This annex specifies the method to be followed for the quantitative determination, by means of a hydrometer, of the particle size distribution of mineral soil material that has been wet-sieved through a 0,063 mm aperture sieve. The analysis requires that the particle density be known or be able to be assumed (see [Clause 4](#)), and that the mass of soil taken for analysis, and the organic carbon content of the sample, be known (Reference [4]). Studies have shown that the hydrometer method is less precise than the pipette method, and that the single greatest error in the former lies in the reading of the hydrometer scale. The hydrometer method shall not be used for samples in which organic matter has not been destroyed, as floating organic matter exacerbates the latter problem.

#### B.2 Apparatus

Items for which details are not otherwise given are as stated in [8.2](#).

##### B.2.1 Hydrometer, of the type illustrated in [Figure B.1](#).

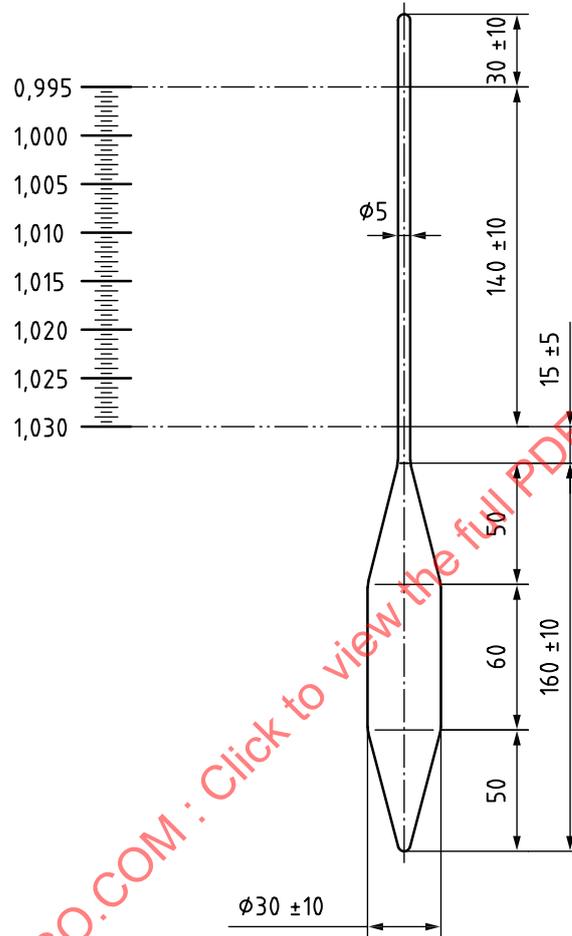
The bulb and stem shall be made from glass free from visible defects, resistant to chemicals and well annealed. Any loading materials fitted to the outside base of the hydrometer shall be affixed with a heat- (up to 80 °C), water- and chemical-resistant cement. The soundness of the fixative shall be verified at least weekly when the hydrometer is in use and repaired if not in good condition. The scale and markings shall be in permanent ink on a high-quality paper with a smooth surface (Esparto paper is a suitable grade). The stem and bulb shall be circular in cross-section and symmetrical about their common long axis. There shall be no abrupt changes of form that will hinder drying and cleaning, or allow the entrapment of air bubbles. The hydrometer shall float, over its whole range, with the stem within 1,5° of the vertical. The graduation lines shall be straight, of uniform thickness, and show no irregularities in spacing. The scale shall be straight, without twist, and with graduation lines at right angles to the long axis of the hydrometer. The graduation lines shall be every 0,000 5 g/ml, in the format shown in [Figure B.1](#). The basis of the scale shall be density (mass per unit volume) in grams per millilitre. The hydrometer shall be calibrated such that, when placed in water at 20 °C, it shows the true density of water at that temperature (see the last paragraph of this subclause). The hydrometer shall be adjusted in relation to a liquid having a surface tension of about 55 mN/m. The maximum permissible scale error shall be plus or minus one scale division, i.e.  $\pm 0,000\ 5\ \text{g/l}$ . The following shall be marked legibly and permanently on the bulb of the hydrometer in a manner that does not obscure any part of the scale:

- a) the basis of the scale, e.g. g/ml at 20 °C (this is the usual basis for commercially available hydrometers);
- b) the maker's name or mark;
- c) a certifying number;
- d) the number of this document.

Any alteration or repair to the hydrometer can change its mass, and hence require its recalibration. The masses of hydrometers shall be verified at least once a month when they are in frequent use. The

mass of a hydrometer shall always be checked when the instrument is brought back into use after an interval. The identifying numbers and masses of hydrometers shall be recorded in a book kept for that purpose. Readings taken from hydrometers used in this document shall be recorded in the following manner. Subtract 1,000 from the scale reading and multiply the remaining number by 1 000 (see the last paragraph of this subclause). Scale readings <1,000 will give negative values, whilst scale readings >1,000 will give positive values.

Dimensions in millimetres



NOTE Calibrations in g/ml at 20 °C.

Figure B.1 — Hydrometer for determination of fine particle size

For example:

Observed reading	Recorded value
0,999 5	-0,5
1,000 0	0,0
1,001 5	+1,5

Older hydrometers are commonly calibrated to read a density of water of 1,000 0 g/ml at 20 °C, rather than the true density of 0,998 2 g/ml (References [4] and [6]). It is strongly recommended that older hydrometers of this type not be used in this document. If there are no others available, then a value of 1,8 shall be added to the observed hydrometer reading  $d'$  (see B.4 and B.5 and Table B.1).

**B.2.2 Two 1 L measuring cylinders**, marked at 1 l volume, about 60 mm in diameter, 360 mm high and fitted with ground-glass stoppers.

It is important that all the cylinders have similar dimensions, as this affects the calibration of the hydrometer (B.2.1). If glass stoppers are unavailable, well-fitting rubber stoppers may be used. The cylinders shall not have pouring lips.

**B.2.3 Thermometer**, to cover the temperature range 0 °C to 50 °C, readable to  $\pm 0,5$  °C.

**B.2.4 Mechanical shaker**, capable of keeping 75 g of soil and 150 ml of water in continuous suspension (see 9.2.6).

**B.2.5 Stop clock**, readable to 1 s.

**B.2.6 Millimetre scale**, such as a steel rule.

**B.2.7 Wash bottle**, and a **100 ml measuring cylinder**.

**B.2.8 Constant-temperature environment** capable of being maintained between 20 °C and 30 °C to an accuracy of  $\pm 0,5$  °C (see 9.2.2 and the last paragraph of 9.2.1). If a water bath is used, it shall be capable of accepting the sedimentation tubes immersed to the 1 L mark.

### B.3 Reagents

All reagents and water shall be of the same quality as specified in the first paragraph of 8.3.

**B.3.1 Solution of a dispersing agent** (see 9.3.2).

**B.3.2 Antifoaming agent** (see 9.3.3).

### B.4 Calibrations and corrections

#### B.4.1 General

The hydrometer is used to determine the (usually small) difference in density between a suspension of particles and the liquid (as a separate sample) in which the particles are suspended, this difference being proportional to the amount of particles in suspension. Because the density difference is small, the hydrometer phrasing read as accurately as possible, and any factors which affect this reading shall be allowed for in calculating the particle size parameters. These factors are considered in B.4.2, B.4.3 and B.4.4, and Reference [4].

#### B.4.2 Volume calibration of the hydrometer

The volume of the hydrometer shall be known, as it affects the amount by which the level of the liquid in the cylinder rises when the hydrometer is inserted. Stand a 1 l measuring cylinder on a smooth, level surface, pour approximately 800 ml of water into the cylinder, and record the reading of the water level. Immerse the hydrometer in the water and record the level again. Record the difference between the two levels as the volume of the hydrometer,  $V_h$ , in millilitres.

#### B.4.3 Scale calibration of the hydrometer

The magnitude of this correction is affected by both the volume of the hydrometer (B.4.2), and the sectional area of the sedimentation cylinder, hence the need for cylinders of similar dimensions (B.2.2). Refer to Figure B.2. Use the steel rule to measure the distance between two graduations on the 1 l measuring cylinder (e.g. 100 ml to 900 ml). Call this distance  $L$  (in millimetres), and the indicated

volume between the graduations  $V_L$  (in millilitres). Measure and record the distance from the lowest calibration mark on the stem of the hydrometer to each of the other major calibration marks,  $d_1, d_2, d_3$ , etc. Measure and record the distance  $d_n$  from the neck of the bulb to the nearest calibration mark. The distances  $H_1, H_2, H_3$ , etc., corresponding to readings  $d_1, d_2, d_3, d_n$ , etc., are equal to the sums  $d_n + d_1, d_n + d_2, d_n + d_3$ , etc. Measure and record the distance,  $h$  (in millimetres), from the neck to the bottom of the bulb, as the height of the bulb. Calculate the effective depths,  $z_1, z_2, z_3$ , etc., corresponding to each of the calibration marks,  $d_1, d_2, d_3, d_n$ , etc., from [Formula \(B.1\)](#):

$$z_1 = H_1 + 1/2(h - V_h L / V_L) \tag{B.1}$$

where

$H_1$  is the length from the neck of the bulb to graduation  $d_1$ , in millimetres;

$h$  is the height of the bulb, in millimetres;

$V_h$  is the volume of the hydrometer bulb, in millilitres;

$L$  is the distance between the graduation marks on the measuring cylinder, in millimetres;

$V_L$  is the indicated volume of the measuring cylinder between the graduations used in the determination of  $L$ , in millilitres.

Plot the relationship between  $z$  and  $d$  as a smooth curve. Record the identification number of the hydrometer and the date of the calibration on the calibration readings and the graph.

NOTE This relationship gives the effective depth of the suspension, the relative density of which is given by the hydrometer reading.

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